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Analysis of Chemical Warfare Agents by GC-MS: First Chemical Cluster CRTI Training Exercise

P.A. D'Agostino, C.R. Jackson Lepage, J.R. Hancock and C.L. Chenier
Defence R&D Canada – Suffield

Technical Memorandum
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Author

Paul A. D'Agostino

Approved by

Jacques Lavigne

H/CBDS

Approved for release by

Robin Clewley

DRP Chair

Abstract

The Chemical Cluster, one of three clusters created by the Chemical, biological, radiological and nuclear Research and Technology Initiative (CRTI), was established to help Canada prepare for possible terrorist events. This working group, made up of representatives from Canadian government departments, has identified a number of chemicals of concern and assigned laboratories with appropriate expertise to provide the analytical support necessary to confirm these compounds in suspect samples. The Royal Canadian Mounted Police (RCMP), in its lead forensics role, will attempt to tentatively identify the chemical(s) of concern and pass on the samples to the responsible laboratory within the Chemical Cluster. Samples containing large amounts of relatively pure chemical warfare agents should trigger a response with one the chemical monitoring devices (e.g., Chemical Agent Monitor) used by the RCMP to triage samples. Defence R&D Canada – Suffield (DRDC Suffield) has been tasked to analyse samples suspected to contain chemical warfare agents for the Chemical Cluster and would receive this type of suspect sample. There remains a possibility that samples with a lower level of chemical warfare agent contamination might inadvertently find their way into a laboratory tasked with another type of analysis. To manage this possibility, the laboratories receiving these types of samples should have an analytical screening capability to allow for the tentative identification of chemical warfare agents in samples and sample extracts. This report summarizes the chemical warfare agent training course in sample preparation and analysis by gas chromatography-mass spectrometry (GC-MS) given by DRDC Suffield to other Chemical Cluster laboratories.

Résumé

Le Groupe chimique, un des trois groupes créés par l'Initiative de recherche et de technologie chimique, biologique, radiologique et nucléaire (IRTC), a été établi pour aider le Canada à se préparer à de possibles évènements terroristes. Ce groupe de travail, composé de représentants de ministères gouvernementaux, a identifié un certain nombre d'agents chimiques inquiétants et a assigné le travail de fournir le soutien analytique nécessaire à la confirmation de ces composés, dans les échantillons suspects, aux laboratoires qui possèdent l'expertise adéquate. La Gendarmerie royale du Canada (GRC), dont le rôle judiciaire est prépondérant, tentera en premier lieu, d'identifier l'agent ou les agents chimiques inquiétants et fera passer les échantillons au laboratoire responsable, à l'intérieur du Groupe chimique. Les échantillons contenant une grande quantité d'agents de guerre chimiques relativement purs devraient déclencher une réaction au moyen d'un des appareils de détection chimique (p. ex : un moniteur d'agent chimique) utilisé par la GRC pour trier les échantillons. R & D pour la défense Canada – Suffield (RDDC Suffield) a reçu du Groupe chimique la mission d'analyser les échantillons suspectés de contenir des agents de guerre chimiques, et recevrait ce type d'échantillon suspect. Il est toujours possible que des échantillons d'agents de guerre chimiques, ayant un plus faible taux de contamination soient dirigés par erreur vers des laboratoires ayant pour mission un différent type d'analyse. Pour gérer cette possibilité, les laboratoires, recevant ces types d'échantillons, devraient posséder une capacité de sélection permettant l'identification préliminaire des agents de guerre chimiques dans des échantillons et des extraits d'échantillons. Ce rapport fait la synthèse du cours de formation portant sur la préparation et l'analyse d'échantillons par couplage chromotographie en phase gazeuse - spectrométrie de masse (CG-MS), donné par RDDC Suffield aux autres laboratoires du Groupe chimique pour identifier les agents de guerre chimiques.

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Executive summary

Introduction: Concerns over possible terrorist use, continued interest by the defence community and the requirements of a verifiable Chemical Weapons Convention (CWC), have driven the development and application of analytical methods for the detection, characterization and confirmation of chemical warfare agents. The Chemical Custer working group within the Chemical, biological, radiological and nuclear Research and Technology Initiative (CRTI) has identified a number of chemicals of concern and assigned laboratories with appropriate expertise to provide the analytical support necessary to confirm these compounds in suspect samples. The Royal Canadian Mounted Police (RCMP), in its lead forensics role, will attempt to tentatively identify the chemical(s) of concern and pass on the samples to the responsible laboratory within the Chemical Cluster. Samples containing large amounts of relatively pure chemical warfare agents should trigger a response with one the chemical monitoring devices (e.g., Chemical Agent Monitor) used by the RCMP to triage samples. Defence R&D Canada – Suffield (DRDC Suffield) has been tasked to analyse samples suspected to contain chemical warfare agents for the Chemical Cluster and would receive this type of suspect sample. There remains a possibility that samples with a lower level of chemical warfare agent contamination might inadvertently find their way into a laboratory tasked with another type of analysis. To manage this possibility, the laboratories receiving these types of samples should have an analytical screening capability to allow for the tentative identification of chemical warfare agents in samples and sample extracts. This report summarizes a three day chemical warfare agent training course in sample preparation and analysis by GC-MS given by DRDC Suffield to other Chemical Cluster laboratories.

Results: The analytical exercise participants successfully analysed a chemical warfare agent test mixture by GC-MS, interpreted the acquired mass spectra and correctly identified the unknown chemical warfare agents spiked into two soil samples. Chemical warfare agents were identified in the soil sample extracts on the basis of both a GC retention time and EI mass spectrometric match with authentic reference standards (or library data).

The analytical participants were briefed on both safety considerations and chemical warfare agent detection devices. Detection devices, including the Chemical Agent Monitor, were demonstrated and sampling kits were available for examination.

Significance: Each of the analytical exercise participants conduct sample handling and analysis for a variety of target compounds for their government departments (Health Canada, Canadian Food Inspection Agency, Royal Canadian Mounted Police and Environment Canada). If their sample handling methods co-extracted chemical warfare agents the analysts would be able to identify the common chemical warfare agents provided the GC-MS analyses were conducted under full scanning EI-MS conditions.

Future Plans: This analytical training exercise may be provided to additional government partners to further their ability to respond to the chemical/biological/nuclear threat.

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Sommaire

Introduction: L'inquiétude au sujet d'une attaque terroriste possible, un intérêt soutenu des organismes de défense et les besoins en objectifs vérifiables de la Convention sur les armes chimiques (CAC) ont conduit à la mise au point et à l'application de méthodes analytiques pour la détection, la caractérisation et la confirmation des agents de guerre chimiques. Le groupe de travail du Groupe chimique, faisant partie de l'Initiative de recherche et de technologie chimique, biologique, radiologique et nucléaire (IRTC), a identifié un certain nombre d'agents chimiques inquiétants et a assigné le travail de fournir le soutien analytique nécessaire visant à confirmer ces composés, dans les échantillons suspects, aux laboratoires qui possèdent l'expertise adéquate. La Gendarmerie royale du Canada (GRC), dont le rôle judiciaire est prépondérant, tentera en premier lieu, d'identifier l'agent ou les agents chimiques inquiétants et fera passer les échantillons au laboratoire responsable à l'intérieur du Groupe chimique. Les échantillons contenant une grande quantité d'agents de guerre chimiques relativement purs devraient déclencher une réaction au moyen d'un des appareils de détection chimique (p. ex : un moniteur d'agent chimique) utilisé par la GRC pour trier les échantillons. R & D pour la défense Canada – Suffield (RDDC Suffield) a reçu du Groupe chimique la mission d'analyser les échantillons suspectés de contenir des agents de guerre chimiques et recevrait ce type d'échantillon suspect. Il est toujours possible que des échantillons d'agents de guerre chimiques, ayant un plus faible taux de contamination soient dirigés par erreur vers des laboratoires ayant pour mission un différent type d'analyse. Pour gérer cette possibilité, les laboratoires recevant ces types d'échantillons devraient posséder une capacité de sélection permettant l'identification préliminaire des agents de guerre chimiques dans des échantillons et des extraits d'échantillons. Ce rapport fait la synthèse d'un cours de formation contre les agents de guerre chimique d'une durée de trois jours, portant sur la préparation et l'analyse d'échantillons par couplage chromotographie en phase gazeuse - spectrométrie de masse (CG-MS), donné par RDDC Suffield aux autres laboratoires du Groupe chimique.

Résultats : Durant les exercices analytiques, les participants ont réussi à analyser un mélange d'essais d'agents de guerre chimique par CG-MS, à interpréter the spectres de masses acquis, et à correctement identifier les agents de guerre chimiques inconnus semés dans deux échantillons de sol. On s'est basé sur le temps de rétention CG ainsi que sur la correspondance entre la masse spectrométrique EI et les normes de références authentiques (les bibliothèques de données) pour identifier les agents de guerre chimiques dans les extraits d'échantillons de sol.

Les participants analytiques ont été informés à la fois au sujet des considérations de sécurité et des appareils de détection des agents de guerre chimiques. Les appareils de détection, dont le moniteur d'agent chimique, ont été démontrés et des trousses d'échantillons étaient disponibles pour examen.

Portée des résultats : Chaque participant aux exercices analytiques a effectué des manipulations d'échantillons et des analyses pour une variété d'éléments visés pour leurs ministères (Santé Canada, l'Agence canadienne d'inspection des aliments, la Gendarmerie royale du Canada et Environnement Canada). Si leurs méthodes de manipulation

d'échantillons réussissent à co-extraire les agents de guerre chimiques, les analystes seront capables d'identifier les agents de guerre chimiques communs dans la mesure ou les analyses CG-SM sont conduites dans les conditions de balayage complet EI-SM.

Plans futurs : Cet exercice de formation analytique pourra être mis à la disposition d'autres partenaires gouvernementaux afin d'améliorer les capacités de réponse à la menace chimique, biologique et nucléaire de ces derniers.

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Table of contents

List of figures

List of tables

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Introduction

CRTI training - Identification of chemical warfare agents

The Chemical Cluster, one of three clusters created by the Chemical, biological, radiological and nuclear Research and Technology Initiative (CRTI), was established to help Canada prepare for possible terrorist events. This working group, made up of representatives from Canadian government departments, has identified a number of chemicals of concern and assigned laboratories with appropriate expertise to provide the analytical support necessary to confirm these compounds in suspect samples. The Royal Canadian Mounted Police (RCMP), in its lead forensics role, will attempt to tentatively identify the chemical(s) of concern and pass on the samples to the responsible laboratory within the Chemical Cluster. Samples containing large amounts of relatively pure chemical warfare agents should trigger a response with one the chemical monitoring devices (e.g., Chemical Agent Monitor) used by the RCMP to triage samples. Defence Research and Development Canada (DRDC Suffield) has been tasked to analyse samples suspected to contain chemical warfare agents for the Chemical Cluster and would receive this type of suspect sample. There remains a possibility that samples with a lower level of chemical warfare agent contamination might inadvertently find their way into a laboratory tasked with another type of analysis. To manage this possibility, the laboratories receiving these types of samples should have an analytical screening capability to allow for the tentative identification of chemical warfare agents in samples or sample extracts.

DRDC Suffield provided a three-day chemical warfare agent training course in sample preparation and analysis by GC-MS. Four "hands-on" analysts from laboratories with GC-MS experience within the Chemical Cluster were provided with both lectures and chemical warfare agent training designed to aid in the tentative identification of chemical warfare agents in collected samples.

Exercise Outline:

1. Lectures on sampling handling and analysis of chemical warfare agents by GC-MS.

2. Analysis of chemical warfare agent standards by GC-MS. Interpretation of MS data.

3. Sample handling and analysis of a soil sample(s) contaminated at the μg/g level (part per million) with chemical warfare agent(s). Interpretation of GC-MS data.

4. Lecture on field detection of chemical warfare agents.

Historical background

Chemical warfare agents are a group of toxic chemicals that have been defined in the Convention on the Prohibition of the Development, Production, Stockpiling and Use of Chemical Weapons and their Destruction (commonly referred to as the Chemical Weapons Convention or CWC) as "any chemical which through its chemical effect on life processes can cause death, temporary incapacitation or permanent harm to humans or animals ...". Poisonous or toxic compounds have been utilized in an effort to gain military superiority throughout history but it is only during the past century that chemical warfare agents have been produced and used on a large scale. Tear gas grenades were used in 1914 by the French at the outbreak of the First World War, but it was not until the Germans first used chlorine near Ypres in 1915 that the world entered the modern era of chemical warfare. Other chemical warfare agents such as phosgene and mustard were weaponized during the First World War and were used by both sides throughout the conflict.

The use and development of chemical warfare agents continued following the First World War despite the signing of the 1925 Geneva Protocol, which bans the first use of chemical weapons. Mustard was used by the Italians against the Abyssinians (Ethiopia) during the 1936-1937 war and just prior to the Second World War, the Germans discovered and produced the first nerve agent, tabun. Tabun was weaponized by the Germans but neither side made use of their chemical weapons stocks. More effective nerve agents, such as VX, were developed in the 1950's, mustard was used in the Yemen Civil War (1963-1967) and allegations of chemical warfare agent use were reported in South East Asian conflicts. Nerve and mustard agents were used by Iraq in the 1980's war between Iran and Iraq, and were considered a real threat to United Nations armed forces during their action against Iraq in1991. Mustard and sarin were detected in samples collected in 1992 from a site where chemical weapons were though to have been previously used against a Kurdish village. Most recently, sarin was released by the Aum Shinrikyo cult in the Tokyo underground transit system (1995) resulting in thousands seeking medical attention and twelve deaths.

After considerable effort, the CWC was opened to signature in 1993, with the treaty coming into force on April 29, 1997. More than 140 State Parties have ratified the CWC and agreed not to develop, produce, stockpile, transfer or use chemical weapons and agreed to destroy their own chemical weapons and production facilities. A strong compliance monitoring regime involving site inspections was built into the CWC to ensure that the treaty remains verifiable. The Organisation for the Prohibition of Chemical Weapons, or OPCW, based in the Hague has responsibility for implementation of the treaty. Routine OPCW inspections have taken place at declared sites, including small-scale production, storage and destruction sites, and challenge inspections will take place at sites suspected of non-compliance. Proliferation of chemical weapons and their use will hopefully decrease over the coming years as the CWC proceeds towards its goal of world-wide chemical weapons destruction.

Recent concerns over possible terrorist use, continued interest by the defence community and the requirements of a verifiable CWC, have driven the development and application of analytical methods for the detection, characterization and confirmation of chemical warfare agents. Analytical techniques play an important role in this process as sampling and analysis will be conducted to ensure treaty compliance, to investigate allegations of use and to verify the use of these weapons for forensic purposes.

Chemical warfare agent categories

Chemical warfare agents have been classified into nerve, blister, choking, vomiting, blood, tear and incapacitating agent categories based on their effect on humans. The most significant chemical warfare agents in terms of military capacity and past use are the nerve and blister agents. For these reasons the analysis of these compounds will be emphasized over the other groups. The choking, blood and vomiting agents are for the most part obsolete chemical agents that were employed during the First World War. The tear agents were used during the Vietnam War but their primary use, because of their inability to produce high casualties, remains in riot control and training. Incapacitating agents have been included in the CWC as the United States did develop an agent in this category.

The compounds listed in Table 1 represent the most common chemical warfare agents, with their Chemical Abstracts registry numbers, and is not intended to be exhaustive. It has been estimated that more than 10,000 compounds are controlled under the CWC, although in practical terms the actual number of chemical warfare agents, precursors and degradation products that are contained in the OPCW database is in the hundreds. The structures of common nerve and blister chemical warfare agents are illustrated in Figure 1.

Table 1. *Common Chemical Warfare Agents*

a) Nerve (reacts irreversibly with cholinesterase which results in acetylcholine accumulation, continual stimulation of the body's nervous system and eventual death)

Full Name (Trivial Name(s))	CAS No.
1-Methylethyl methylphosphonofluoridate (sarin, GB)	107-44-8
1,2,2-Trimethylpropyl methylphosphonofluoridate (soman, GD)	96-64-0
Cyclohexyl methylphosphonofluoridate (GF)	329-99-7
Ethyl dimethylphosphoramidocyanidate (tabun, GA)	77-81-6
O-Ethyl S-(2-diisopropylaminoethyl) methylphosphonothiolate (VX)	50782-69-9

b) Blister (affects the lungs, eyes and produces skin blistering)

Full Name (Trivial Name(s))	CAS No.
Bis(2-chloroethyl)sulfide (mustard, H)	505-60-2
Bis(2-chloroethylthio)ethane (sesquimustard, Q)	3563-36-8
Bis(2-chloroethylthioethyl)ether (T)	63918-89-8
Tris(2-chloroethyl)amine (HN-3)	555-77-1
(2-chloroethenyl)arsonous dichloride (lewisite, L)	541-25-3

c) Choking (affects respiratory tract and lungs)

Full Name (Trivial Name(s))	CAS No.
Chlorine	7782-50-5
Phosgene (CG)	75-44-5

d) Vomiting (causes acute pain, nausea and vomiting in victims)

Full Name (Trivial Name(s))	CAS No.
Diphenylarsinous chloride (DA)	712-48-1
10-Chloro-5,10-dihydrophenarsazine (adamsite, DM)	578-94-9
Diphenylarsinous cyanide (DC)	23525-22-6

e) Blood (prevents transfer of oxygen to the body's tissues)

Full Name (Trivial Name(s))	CAS No.
Hydrogen cyanide (HCN, AC)	74-90-8

f) Tear (causes tearing and irritation of the skin)

Full Name (Trivial Name(s))	CAS No.
[(2-chlorophenyl)methylene]propanedinitrile (CS)	2698-41-1
2-Chloro-1-phenylethanone (CN)	532-27-4
Dibenz[b,f][1,4]oxazepin (CR)	257-07-8

g) Incapacitating (prevents normal activity by producing mental or physiological effects)

Full Name (Trivial Name(s))	CAS No.
3-Quinuclidinyl benzilate (BZ)	6581-06-2

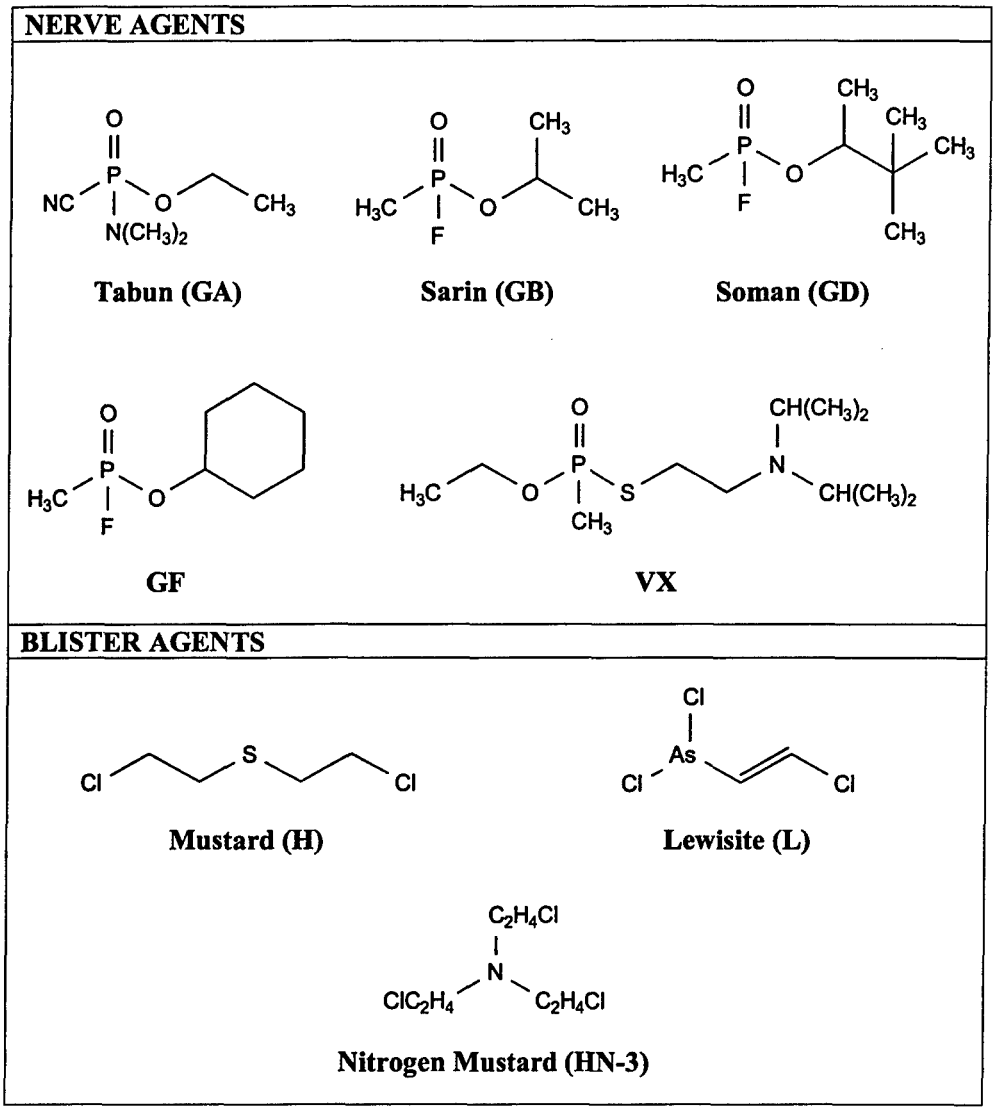

Figure 1. *Structures of common chemical warfare agents.*

Identification methods

Chemical warfare agents have often been referred to as warfare gases and, the military phrase "gas, gas, gas" has become synonymous with attack by chemical warfare agents. In fact, many chemical warfare agents exist as liquids at ambient temperatures but have varying degrees of volatility and pose both a vapor hazard as well as a liquid contact hazard. This physical characteristic has made the analysis of chemical warfare agents amenable to the analytical techniques commonly employed for most environmental analyses, namely gas

chromatography (GC) and liquid chromatography (LC) with a variety of detectors including mass spectrometry (MS). Synthetic or relatively pure samples not requiring chromatographic separation are also frequently characterized by nuclear magnetic resonance (NMR) or Fourier transform infrared (FTIR) spectroscopy.

The OPCW inspectorate, an important end user of analytical techniques for chemical warfare agents, requires the use of two or more spectrometric techniques and the availability of authentic reference standards for the unambiguous identification of controlled compounds. For this reason, the combined use of GC-FTIR has received increased attention as newer technologies have led to detection limits approaching those routinely reported during GC-MS analysis. For analyses involving low levels of chemical warfare agents in the presence of high levels of interfering chemical background, tandem mass spectrometry (MS/MS) is often employed.

Chromatography

Samples contaminated with chemical warfare agents typically contain multiple components that are best characterized following chromatographic separation. These samples generally fall into one of the following general categories; a) munitions or munition fragments (e.g., neat liquid or artillery shell casing), b) environmental (e.g., soil, water, vegetation or air samples), c) man-made materials (e.g., painted surfaces or rubber) and d) biological media (e.g., blood or urine). The ease of analysis depends on the amount of sample preparation required to obtain a suitable sample or extract for chromatographic analysis. In the simplest case where neat liquid can be obtained, the sample requires dilution with a suitable solvent prior to analysis. Environmental and other samples generally require (at a minimum) solvent extraction and concentration prior to analysis.

Capillary column GC is the most frequently employed analytical separation method for the screening of samples contaminated with chemical warfare agents. Separation of chemical warfare agents may be achieved with many of the commercially available fused silica columns coated with polysiloxane or other films and retention index data relative to n-alkanes and alkylbis(trifluoromethyl)phosphine sulfides (M-series) have been reported for many chemical warfare agents and related compounds. In general, the best separations have been achieved with moderately polar films such as (86%)-dimethyl-(14%)-cyanopropylphenyl-polysiloxane. Chiral stationary phases have also been developed for the resolution of stereoisomers of several chiral nerve agents, most notably soman. The use of multiple columns of differing polarity during one analysis has been successfully employed during chemical warfare agent analysis and the term "retention spectrometry" was coined to describe this technique.

Most of the GC detectors commonly applied to pesticide residue analysis have also been applied to the screening of samples for chemical warfare agents with detection limits typically being in the nanogram to picogram range. Flame ionization detection (FID) is routinely used for preliminary analyses as this technique provides a good indication of the complexity of a sample extract. Figure 2 illustrates typical GC-FID chromatographic separations obtained for three different munitions-grade mustard formulations, HT, HS and HQ, each of which contain

mustard and a number of related longer chain blister agents. The longer chain blister agents, sesquimustard (Q) and bis[(2-chloroethylthio)-ethyl]ether (T) were present in all three samples along with a number of other related compounds that may provide synthetic procedure or source information.

The need for higher specificity and sensitivity has led to the application of element specific detectors such as flame photometric detection (FPD), thermionic detection (TID), atomic emission (AED) and electron capture detection (ECD). The simultaneous use of FID with one or more element specific detectors has also been demonstrated during dual or tri channel GC analysis using conventional and thermal desorption sample introduction. While data obtained with these detectors may provide strong collaborative evidence for the presence of chemical warfare agents, they cannot be used for full confirmation. Use of GC with one or more spectrometric technique ssuch as MS is required to confirm the presence of chemical warfare agents.

Both the nerve and blister agents undergo hydrolysis in the environment and methods are required under the Chemical Weapons Convention for retrospective detection and confirmation of these compounds. These compounds are significant as they would not be routinely detected in environmental samples and their identification strongly suggest the prior presence of chemical warfare agents. The degradation products of the chemical warfare agents, in particular the nerve agents, are non-volatile hydrolysis products that must be derivatized prior to GC analysis. A variety of derivatization reagents, leading to the formation of pentfluorobenzyl, methyl, *tert*-butyldimethylsilyl and trimethylsilyl ethers (or esters), have been investigated to allow GC analysis of organophosphorus acids related to the nerve agents (e.g., alkyl methylphosphonic acids and methylphosphonic acid). Increasingly, LC-ESI-MS is being used for these types of analyses, as electrospray mass spectrometric data may be used to identify chemical warfare agents, their degradation products and related compounds in aqueous samples or extracts without the need for additional sample handling and derivatization steps.

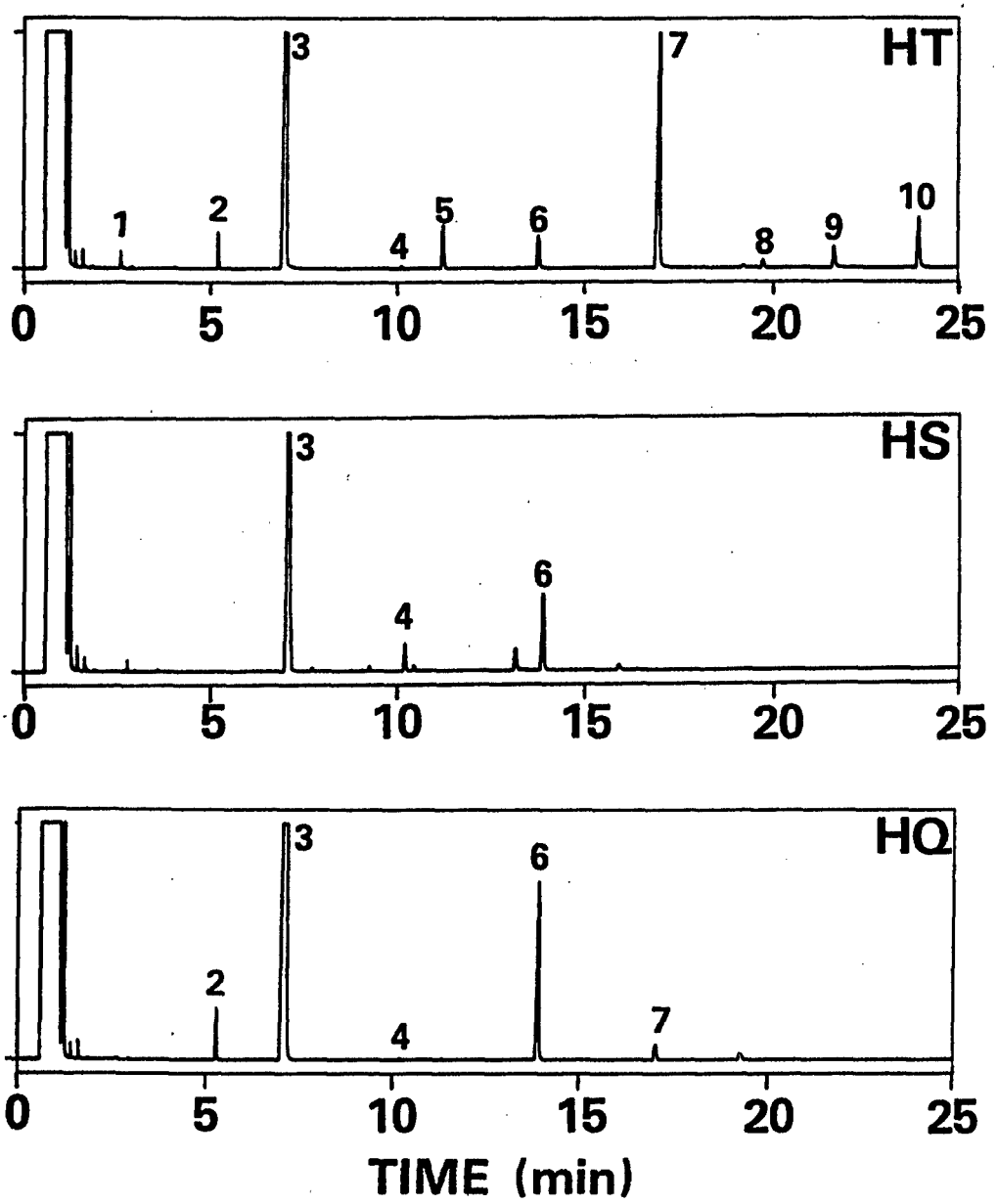

Figure 2. *Capillary column GC-FID chromatograms of three munitions- grade mustard samples; HT (top), HS (middle) and HQ (bottom). Identified compounds include: 1. 1,4-thioxane, 2. 1,4-dithiane, 3. mustard (H), 4. bis(2-chloroethyl)disulfide, 5. 2-chloroethyl (2-chloroethoxy)ethyl sulfide, 6. sesqui-mustard (Q), 7. bis(2-chloroethylthioethyl)ether (T), 8. 1,14-dichloro-3,9-dithia-6,12-dioxatetradecane, 9. 1,14-dichloro-3,6,12-trithia-9-oxatetradecane and 10. 1,16-dichloro-3,9,15-trithia-6,12-dioxaheptadecane. (GC conditions: 15 m x 0.32 mm ID J&W DB-1; 50°C (2 min) 10°C/min 280°C (5 min)).*

Mass spectrometry

Mass spectrometry is the method of choice for the detection and characterization of chemical warfare agents, their precursors, degradation products and related compounds. Extensive use has been made of GC-MS and the mass spectra of numerous chemical warfare agents and related compounds have been published, with the most common chemical warfare agent mass spectra being available in the OPCW, commercial or defence community databases.

Most of the MS data has been obtained under electron impact (EI) ionization conditions. However many of the chemical warfare agents, in particular the organophosphorus nerve agents and the longer chain blister agents related to mustard, do not provide molecular ion information under EI-MS. This hinders confirmation of these chemical warfare agents and makes identification of novel chemical warfare agents or related impurities difficult. For this reason, considerable effort has been devoted to the use of chemical ionization (CI) as a complementary ionization technique. This milder form of ionization generally affords molecular ion information for the chemical warfare agents and has been used extensively for the identification of related compounds or impurities in chemical warfare agent munition samples and environmental sample extracts. The characterization of these related compounds remains important during OPCW or other analyses since this data may provide an indication of the origin of the sample, the synthetic process utilized or the degree of sample degradation (weathering).

Isobutane, ethylene and methane gases were initially demonstrated as suitable CI gases for the acquisition of organophosphorus nerve agent molecular ion information. More recently, the efficacy of ammonia CI-MS for organophosphorus nerve agents and related compounds was demonstrated and many laboratories now employ this complementary confirmation technique. Ammonia CI not only offers abundant molecular ion data but also affords a high degree of specificity as less basic sample components are not ionized by the ammonium ion. Additional structural data may be obtained through the use of deuterated ammonia CI, as this technique provides hydrogen/deuterium exchange data that indicates the presence of exchangeable hydrogen(s) in CI fragmentation ions. Finally, for full confirmation, the acquired EI and CI mass spectrometric data should be compared to authentic reference data obtained under identical experimental conditions.

Figure 3 illustrates EI and ammonia CI data obtained for VX and a significant VX degradation product, bis[2-(diisopropylamino)ethyl] disulfide. The acquired EI data for both compounds, as well as other VX related compounds, are remarkably similar. Both compounds lack a molecular ion and contain a base ion at m/z 114 due to $(CH_2N(iPr)_2)^+$ and additional ions related to the $-SC_2H_4N(iPr)_2$ substituent. Under ammonia CI conditions, mass spectra containing pseudo-molecular and CI fragmentation ions were acquired, with this data being used to confirm molecular mass and differentiate VX related compounds that exhibit similar EI data.

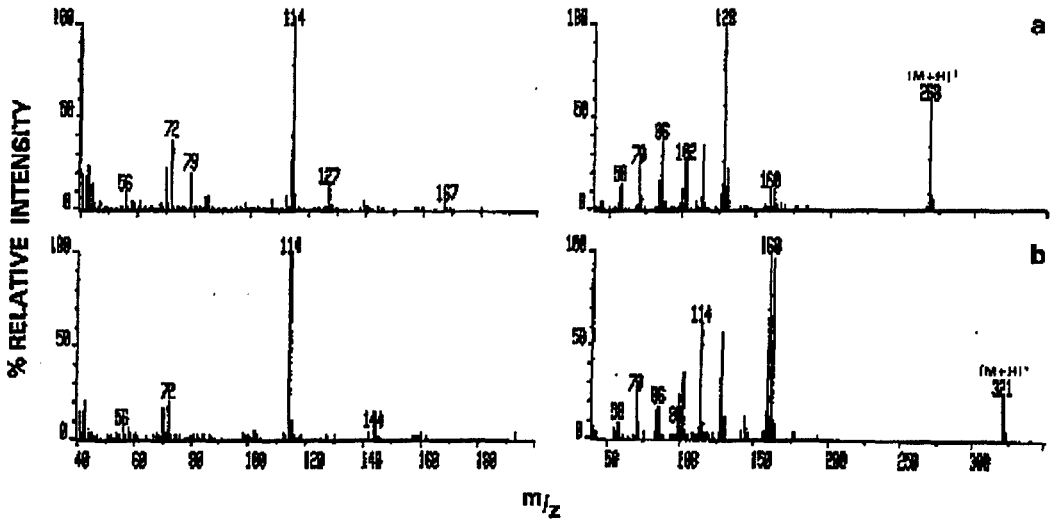

Figure 3. *EI (left) and ammonia CI (right) mass spectrometric data obtained for a) VX and b) bis[2-(diisopropylamino)ethyl] disulfide.*

Capillary column GC-MS/MS offers the analyst the potential for highly specific, sensitive detection of chemical warfare agents as this technique significantly reduces the chemical noise associated with complex biological or environmental sample extracts. The specificity of product scanning with moderate sector resolution, as well as the specificity of ammonia CI, were demonstrated with a hybrid tandem mass spectrometer during analysis of painted panel samples circulated during an international round robin verification exercise.

The painted panel extract was contaminated with numerous hydrocarbons and only two of the three longer chain blister agents, sesquimustard (Q) and bis(2-chloroethylthioethyl)ether (T), could be identified during capillary column GC-MS (EI) analysis (Figure 4a). The arrow indicates the chromatographic retention time of the third blister agent, 2-chloroethyl (2-chloroethoxy)ethyl sulfide (O). The specificity of ammonia CI (Figure 4b) was clearly demonstrated during this analysis. All three longer chain blister agents were identified in the presence of high levels of interfering hydrocarbons, as the hydrocarbons were not sufficiently basic to ionize. Similarly, it was possible to use the resolution of hybrid tandem mass spectrometry to discriminate between ions at m/z 123 arising from the longer chain blister agents from those ions at m/z 123 arising from the hydrocarbon background. The resultant GC-MS/MS chromatogram (Figure 4c), where only m/z 123 ions due to the blister agents were transmitted into the collisional activated dissociation cell, was virtually free of chemical noise and all three components were detected. The three longer chain blister agents were well resolved with the J&W DB-1701 capillary column, with all three components exhibiting similar product spectra during GC-MS/MS analysis.

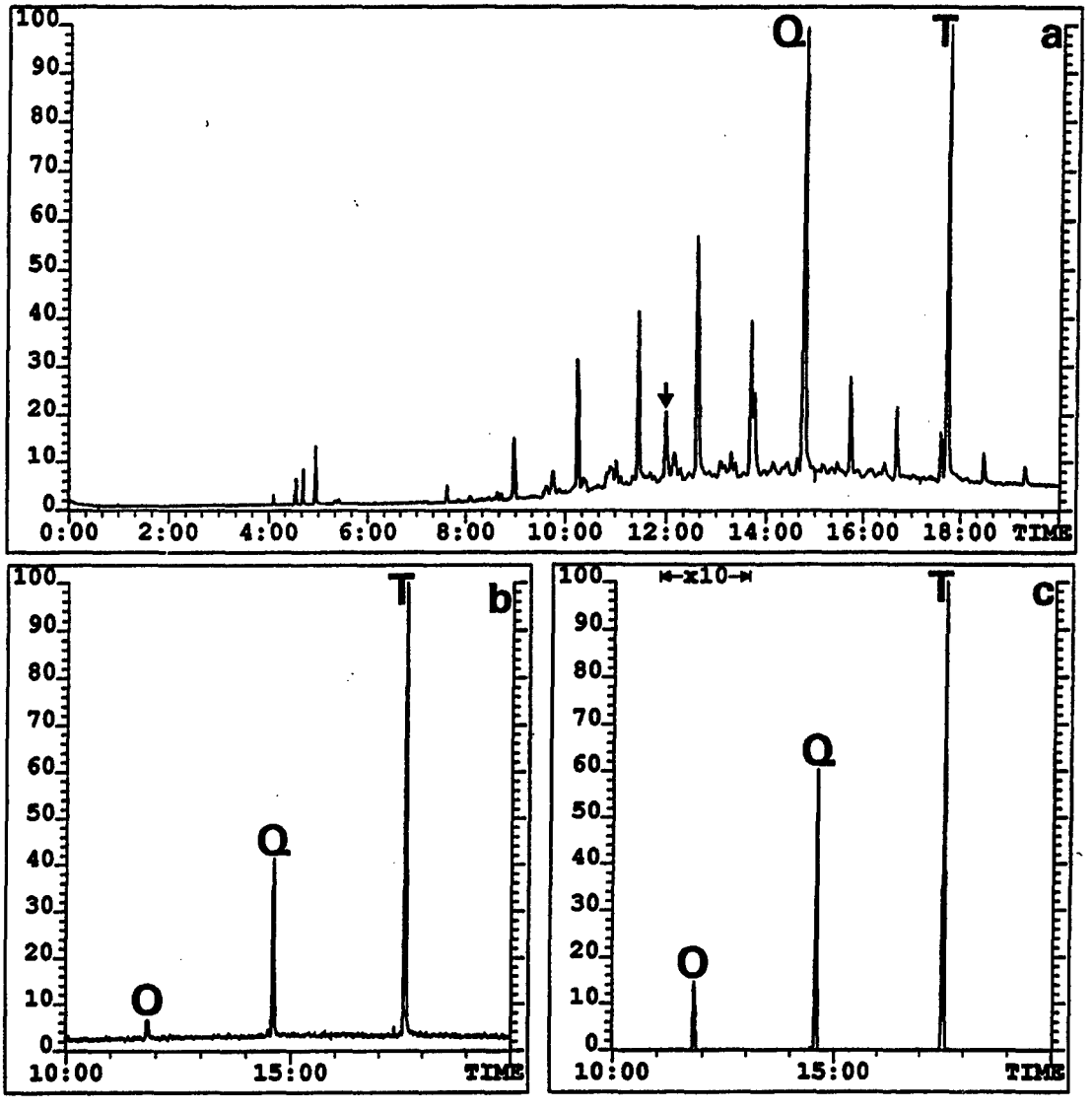

Figure 4. *Capillary column a) GC-MS (EI), b) GC-MS (ammonia CI) and c) GC-MS/MS (EI) chromatograms obtained during analysis of international round robin painted panel extracts. Sequimustard (Q) and bis(2-chloroethylthioethyl)ether (T) were detected during EI analysis. The downward arrow in a) indicates the retention time of 2-chloroethyl (2-chloroethoxy)ethyl sulfide (O). This compound was masked by the sample matrix during EI analysis and was only detected following b) ammonia CI and c) MS/MS analysis. (GC conditions: 15 m x 0.32 mm ID J&W DB-1701, 40°C (2 min) 10°C/min 280°C (5 min), X-axis: time (minutes)).*

Both the nerve and blister agents undergo hydrolysis in the environment and methods are required for retrospective detection and confirmation of these hydrolysis products. Hydrolysis products are significant as they are generally compounds that would not be routinely detected in environmental samples and their presence strongly suggest the prior presence of chemical warfare agents. The degradation products of the chemical warfare agents, in particular the nerve agents, are non-volatile hydrolysis products that must be derivatized prior to GC analysis. Alternatively aqueous samples or extracts may be analyzed by LC-MS, negating the need for additional sample handling steps and derivatization.

Use of thermospray mass spectrometry and more recently the atmospheric pressure ionization (e.g., electrospray (ESI), ionspray and atmospheric pressure CI) techniques has enabled the direct mass spectrometric analysis of the hydrolysis products of chemical warfare agents. These techniques may be interfaced to liquid chromatography for component separation, with thermospray having been largely superceded by atmospheric pressure ionization (API) for most LC-MS applications. LC-ESI-MS methods have been used for the direct analysis of chemical warfare agent hydrolysis products in a number of studies and have recently been used for the analysis of nerve agents. These new methods complement existing GC-MS methods for the analysis of chemical warfare agents and their hydrolysis products and LC-ESI-MS methods will replace some GC-MS methods used for the analysis of contaminated aqueous samples or extracts.

Mustard and longer chain blister agents hydrolyze to their corresponding diols, with thiodiglycol being the product formed following hydrolysis of mustard. Figure 5a illustrates a typical LC-ESI-MS chromatogram obtained for the aqueous extract of a soil sample taken from a former mustard storage site. The soil sample extract contained thiodiglycol (Figure 5b) and 6-oxa-3,9-dithia-1,11-undecanediol (Figure 5c), the hydrolysis products of blister agents mustard and bis(2-chloroethylthioethyl)ether, respectively. ESI-MS data for both compounds contained protonated molecular ions that could be used to confirm molecular mass and characteristic lower mass product ions.

Figure 6 illustrates the LC-ESI-MS chromatogram for a complex munitions-grade tabun sample. Tabun and a number of related compounds were identified based on their acquired ESI-MS data. The mass spectra contained $(M+H)^+$, $(M+H+ACN)^+$ ions and/or protonated dimers that could be used to confirm the molecular mass of each compound. Structural information was provided by inducing product ion formation in either the ESI interface or the quadrupole collisional cell of a MS/MS instrument. Product ions due to alkene loss from the alkoxy substituents, and the acetonitrile adduct associated with these product ions, were generally observed. Figure 7 illustrates typical ESI-MS data obtained for tabun and three other nerve agents.

Considerable effort has been expended on the development of field portable MS and GC-MS instruments, as this technique holds the greatest promise for the confirmation of chemical warfare agents under field situations. The OPCW has available field portable GC-MS instrumentation that may be taken on-site to confirm the presence of chemical warfare agents. An atmospheric pressure MS/MS has also been developed and evaluated for real-time detection of nerve agents in air. Alternatively, air samples may be collected on Solid Phase Microextraction (SPME) fibres or on Tenax tubes that may be thermally desorbed into an on-site GC-MS instrument. Secondary ion mass spectrometry has been used for the detection of

chemical warfare agents and their hydrolysis products on leaves, soil and concrete, offering a new option for the detection of these compounds on adsorptive surfaces. Finally, rapid separation and detection of chemical warfare agents has recently been demonstrated with ESI-ion mobility spectrometry (IMS)-MS. IMS is commonly employed in military devices for rapid field detection and this approach could be lead to the development of instrumentation for the analysis of aqueous samples.

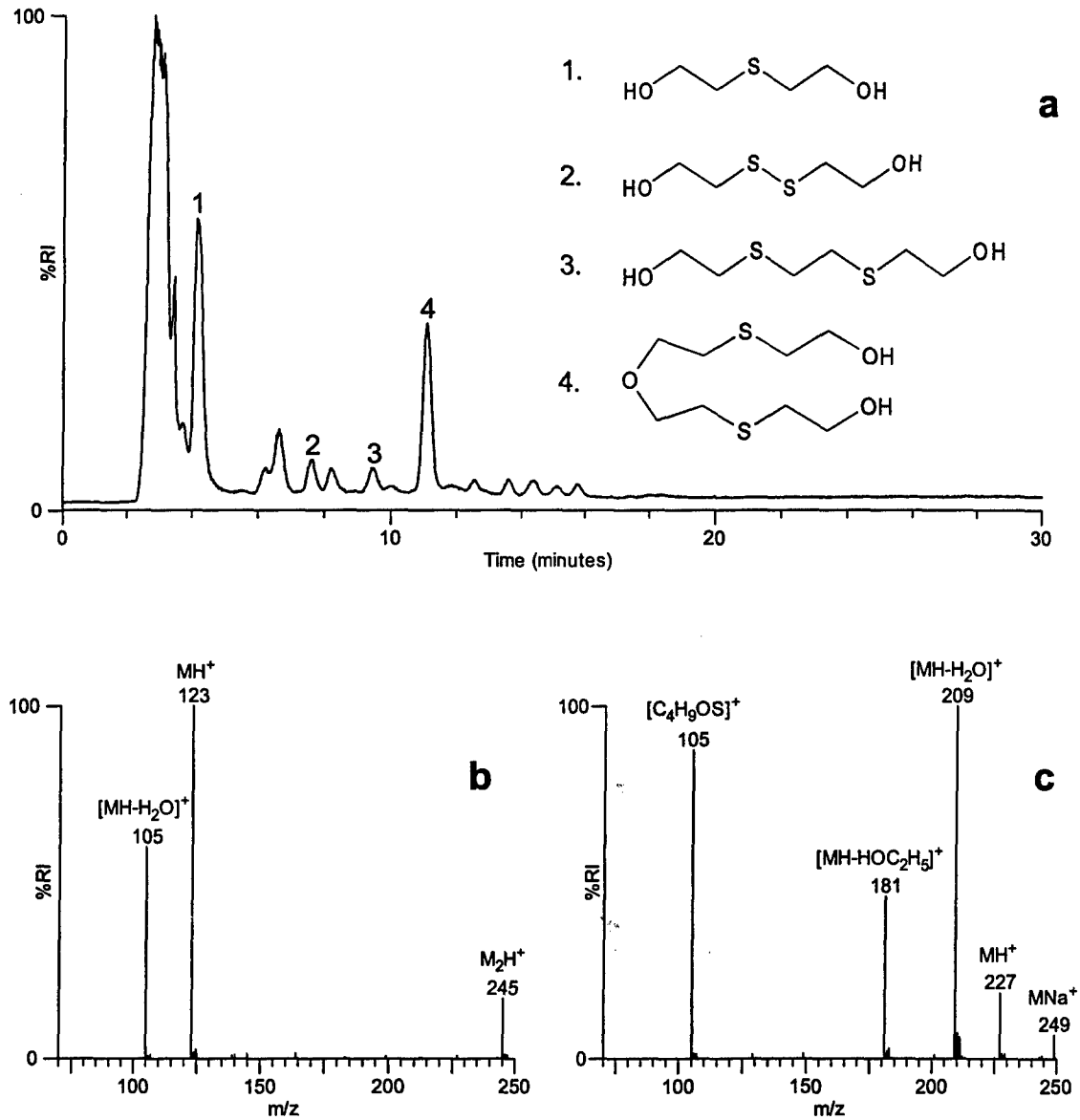

Figure 5. *a) Packed capillary LC-ESI-MS chromatogram obtained for the water extract of a soil sample obtained from a former mustard site. ESI-MS data obtained for b) thiodiglycol (sampling cone voltage: 20 V) and c) 6-oxa-3,9-dithia-1,11-undecanediol (sampling cone voltage: 30 V). (LC condtions: 150 mm x 0.32 mm i.d. C_{18}, acetonitrile/water gradient).*

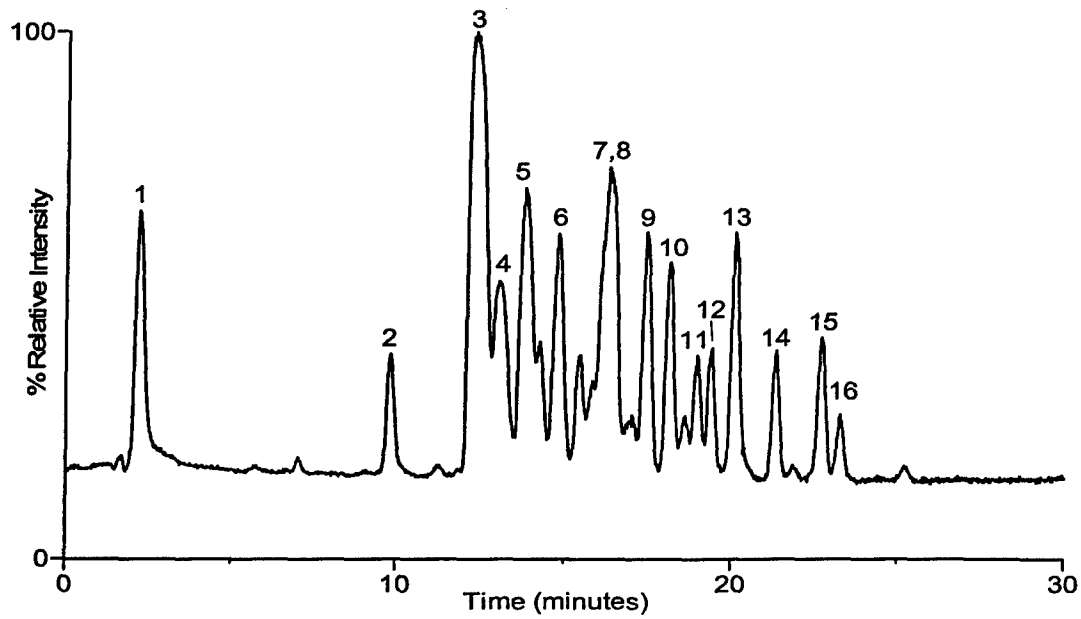

Figure 6. *Packed capillary LC-ESI-MS chromatogram obtained for 0.1 mg/mL munitions-grade tabun sample. Tabun (peak number 3) and fifteen related organophosphorus compounds were identified by ESI-MS. (LC condtions: 150 mm x 0.32 mm i.d. C_{18}, acetonitrile/water gradient).*

Other methods

NMR is an important technique for the structural analysis and characterization of chemical warfare agents, particularly for the authentication of reference materials or unknown chemical warfare agents and related compounds. The presence of heteronucleii such as ^{31}P and ^{19}F in the nerve agents leads to diagnostic splitting patterns and coupling constants due to ^{1}H-^{31}P and ^{1}H-^{19}F spin-spin coupling. The utility of NMR for analysis of complex sample mixtures or for trace analysis is somewhat limited. Specific heteronuclear experiments such as ^{31}P NMR may be used to identify organophosphorus nerve agents in complex matrices. Characteristic chemical shifts of compounds containing a phosphorus-carbon bond and splittings due to phosphorus-fluorine spin-spin coupling can used to screen for the presence of nerve agents. However, ^{31}P chemical shifts are sensitive to temperature, concentration, and solvent and the identification must be supported with additional spectrometric data such as MS. Two-dimensional correlation experiments have been used to help in structural elucidation of unknowns in contaminated samples, making NMR a valuable technique to be used alongside other spectrometric techniques.

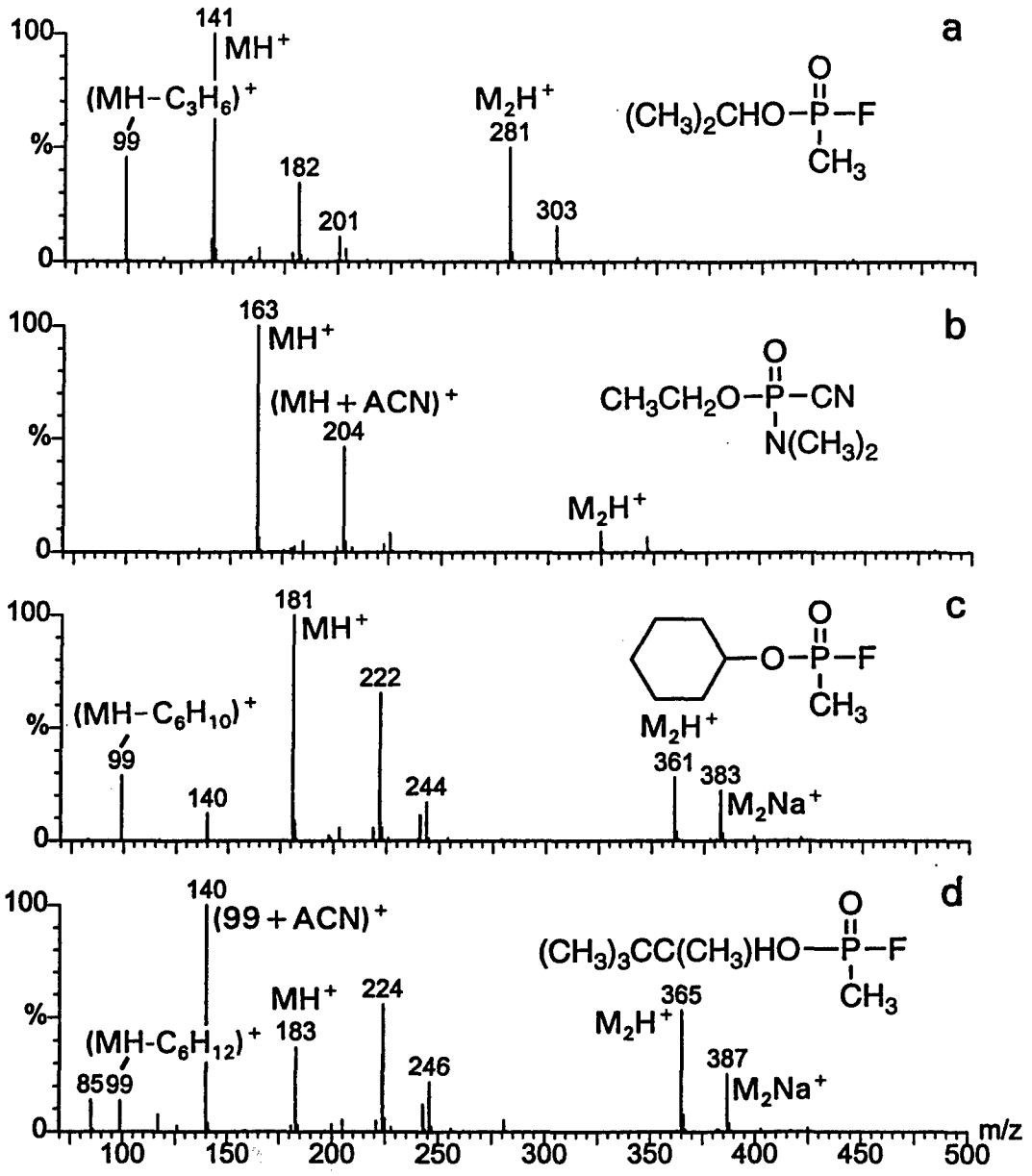

Figure 7. *ESI-MS data obtained for a) sarin (GB), b) tabun (GA), c) cyclohexyl methylphosphonofluoridate (GF) and d) soman (GD) with a sampling cone voltage of 20 volts.*

Condensed phase infrared (IR) data exists for many chemical warfare agents and related compounds as this technique was routinely used prior to the advent of GC-MS. Capillary column GC-FTIR offers considerably more promise for the identification and characterization of chemical warfare agents in multiple component sample extracts and has been utilized as a complementary confirmation technique. Sensitivity is generally poorer than that obtained by

mass spectrometry but may be improved by using large volume (e.g., 50 µL) injections with peak compression onto an uncoated pre-column with lightpipe technology or through the use of cryodeposition.

Military detection

A variety of detection devices and other chemical warfare agent defence equipment have been developed for specific military applications. Most of the effort in this area resulted from the perceived threat during the Cold War era and although this threat has decreased dramatically, interest in chemical detection equipment persists because of world-wide chemical weapons proliferation. During the 1990-1991 Iraq War chemical detection equipment was deployed into the Persian Gulf and similar equipment has been used to support the United Nations Special Commission during the destruction of Iraqi chemical weapons. Equipment of this type has been used by the OPCW and could potentially be utilized again by the United Nations in peacekeeping or intervention roles where the threat of chemical weapons use exists. Table 2 lists examples of chemical detection equipment by country and indicates the principle of detection and capabilities of each system (refer to Jane's Nuclear, Biological and Chemical Defence for a more complete summary).

Safety and disposal

Chemical warfare agents are extremely hazardous and lethal compounds. They should only be used in designated laboratories by personnel trained in safe-handling and decontamination procedures and with immediate access to medical support. Safety and standard operating procedures must be developed and approved before any chemical warfare agents are handled. Chemical warfare agents should only be used in laboratory chemical hoods with a minimum face velocity of 150 linear feet per minute that are equipped with emission control devices that limit exhaust concentration to below 0.0001 mg/m^3. Personnel handling chemical warfare agents should wear rubber gloves, lab coats, and full-faceshields and keep a respirator (gas mask) within easy reach. Sufficient decontaminant to destroy all of the chemical warfare agent being handled must be on hand before commencing operations.

Blister and nerve agents can be destroyed using saturated methanolic solutions of sodium or potassium hydroxide. Decontaminated chemical warfare agents must be disposed of in an environmentally approved method according to local legislation.

Table 2. *Selected Military Chemical Warfare Agent Detection Devices*

Country	Device Name and Capabilities
Canada	Chemical Agent Detection System (CADS II) - Early warning system that controls a network of Chemical Agent Monitors (see U.K.) for the real time detection of nerve and blister agents
China	Chemical Warfare Agent Identification Kit, M-75 - Wet chemistry detection of nerve, blister, choking vomiting and blood agents
Denmark	INNOVA 1312 Multi-Gas Monitor - Photo-acoustic detection of nerve, blister, choking and blood agents
Finland	Chemical Agent Detection System, M90 - Alarm for the ion mobility spectrometric detection of nerve and blister agents
France	PROENGIN Portable Chemical Contamination Monitor AP2C - Hand-held flame photometric detection of nerve and blister agents Also designs for fixed sites (AP2C-V and ADLIF)
Germany	MM-1 Mobile Mass Spectrometer -Quadrupole mass spectrometric detection of chemical warfare agents
	Rapid Alarm and Identification Device – 1 (RAID-1) - Ion mobility spectrometric detection of nerve and blister agents
Switzerland	IMS 2000 CW Agent Detector - Ion mobility spectrometric detection of nerve and blister agents
CIS (formerly USSR)	Automatic Nerve Agent Detector Alarm, Model GSP-11 - Enzyme inhibition for the detection of nerve agents
U.K.	Chemical Agent Monitor (CAM), GID-2/GID-3 Detectors - Ion mobility spectrometry based monitor for the detection of nerve and blister agents
	NAIAD - Nerve agent immobilized enzyme detector and alarm
U.S.A	ICAD Miniature Chemical Agent Detector -Personal detector based on electro-chemical principals for the detection of nerve, blister, blood and choking agents
	MINICAMS - Gas chromatographic detection of nerve and blister agents.
	M21 Remote Sensing Chemical Agent Alarm (RSCAAL) - Passive infrared detection of chemical warfare agents
	Chemical Agent Detection Kit, M256A1 - Wet chemistry detection of nerve, blister, choking and blood agents
	SAW MINICAD MK II - Surface acoustic wave detection of nerve and blister agents

Experimental

Sample and sample handling

Chemical warfare agent standard solutions and contaminated soil samples used in the exercise were prepared and provided by the DRDC Suffield Analytical Laboratory.

The chemical warfare agent test mixture used for quality control purposes contained GB, GD H, GA, GF at the 0.005 mg/mL (in dichloromethane).

Contaminated soil samples were prepared by adding 10 µL of 10 mg/mL GF (in dichloromethane) or 50 µL of 2 mg/mL munitions grade mustard (HQ in dichloromethane) to 2 g of Ottawa sand. The samples were allowed to stand for 1 hour prior to sample handling and analysis by the participants.

Each spiked and control soil sample was ultrasonically extracted for 10 minutes with 4 mL dichloromethane in a 15 x 125 mm screw-capped Teflon-lined glass culture tube. A gross separation of the dichloromethane layer from the soil was performed by centrifugation at 2000 rpm for 10 minutes. An aliquot of the dichloromethane layer (0.4 mL) was removed and centrifuged at 10000 rpm to remove fines, with a portion of this extract being removed and stored in a screw-capped Teflon-lined 1.8 mL sample vial prior to GC-MS analysis.

Instrumental analysis

The dichloromethane extracts were analysed by GC-MS (Agilent 5973N under EI conditions: 70 eV, 0.035 mA, 230°C) using a 15m x 0.25mm ID J&W DB-35MS capillary column and the following temperature program: 40°C (2 min) 10°C/min 280°C (5 min). All injections (1 µL) were cool on-column at 43°C. The mass spectrometer was scanned from 40 to 400 u at 2.08 scans/sec (unit resolution).

Results and discussion

GC-MS analysis of test mixture

A test mixture containing five common chemical warfare agents (GB, GD, GA, H and GF) at the 0.005 mg/mL level was initially analysed to assess the quality of the GC-MS data being generated, to provide an opportunity for handling of a dilute solutions containing chemical warfare agents, and to provide an opportunity to interpret the resultant mass spectra. Figure 8 illustrates a typical GC-MS chromatogram obtained for a 1 μL injection of the chemical warfare agent test mixture. Each sample component (5 ng) was readily resolved and EI mass spectra for each sample component were acquired, interpreted and compared to library mass spectra contained in the EI database supplied with the GC-MS instrument. Figures 9 and 10 illustrate typical EI mass spectra acquired for GB, GD and GF, and H and GA, respectively. The acquired mass spectra compared favorably to those contained in the EI database.

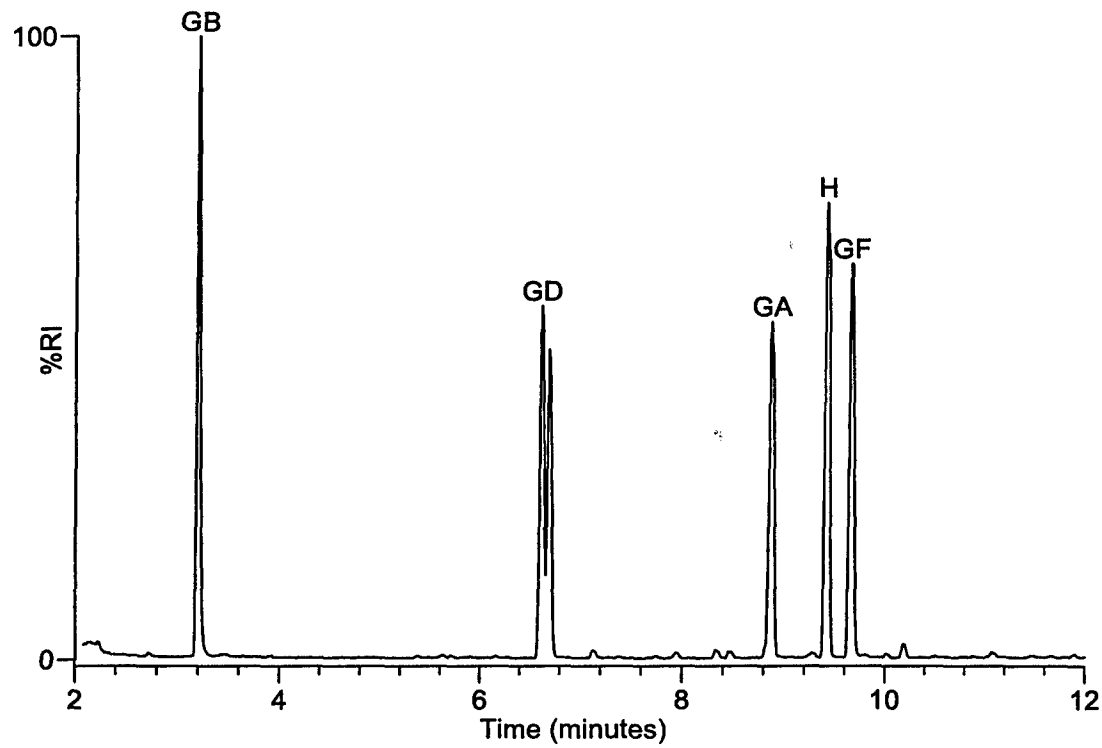

Figure 8. *GC-MS total-ion-current chromatogram of chemical agent test mixture containing 5 ng of GB, GD, GA, H and GF.*

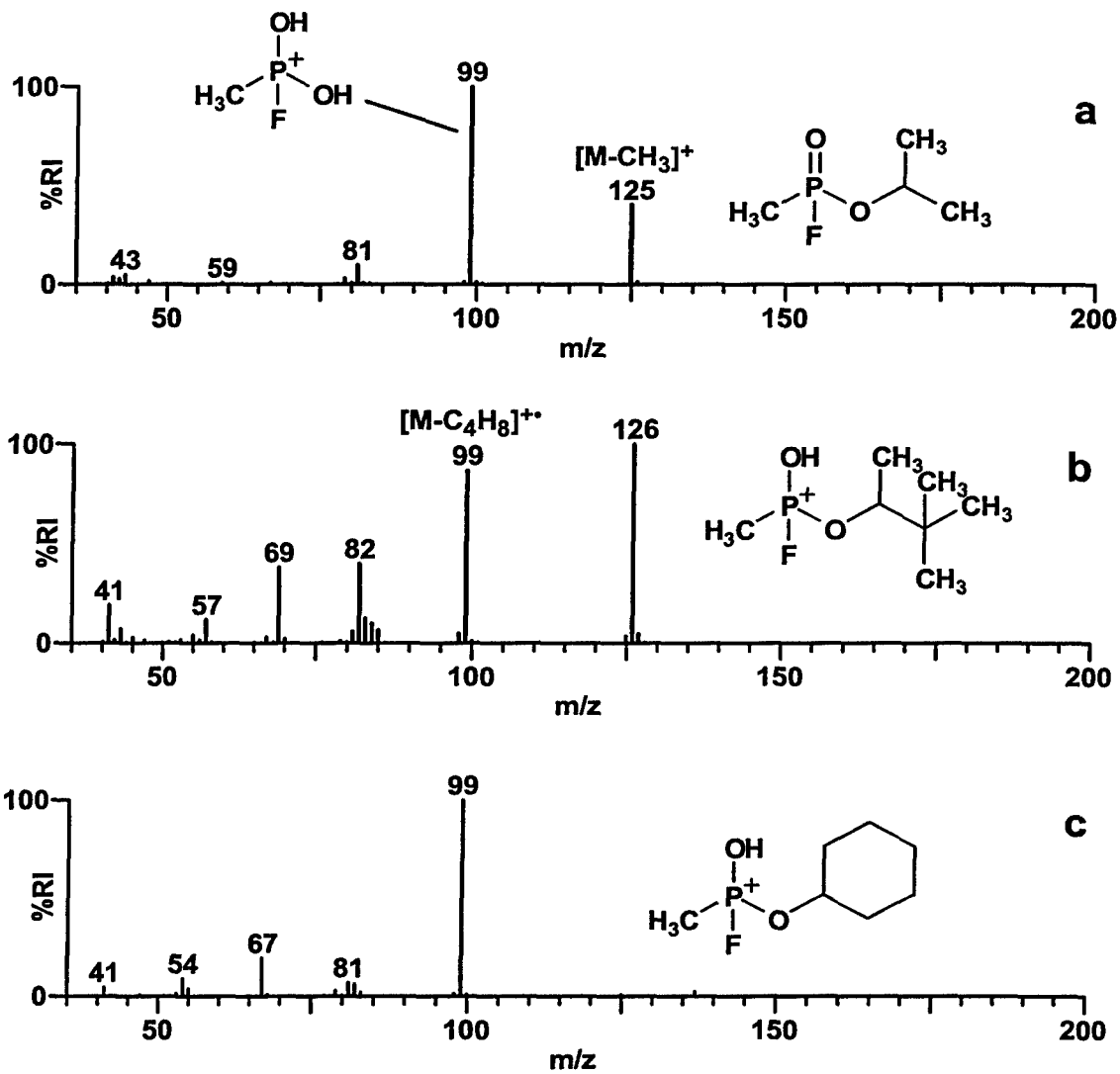

Figure 9. *EI-MS data acquired for a) GB, b) GD and c) GF during GC-MS analysis.*

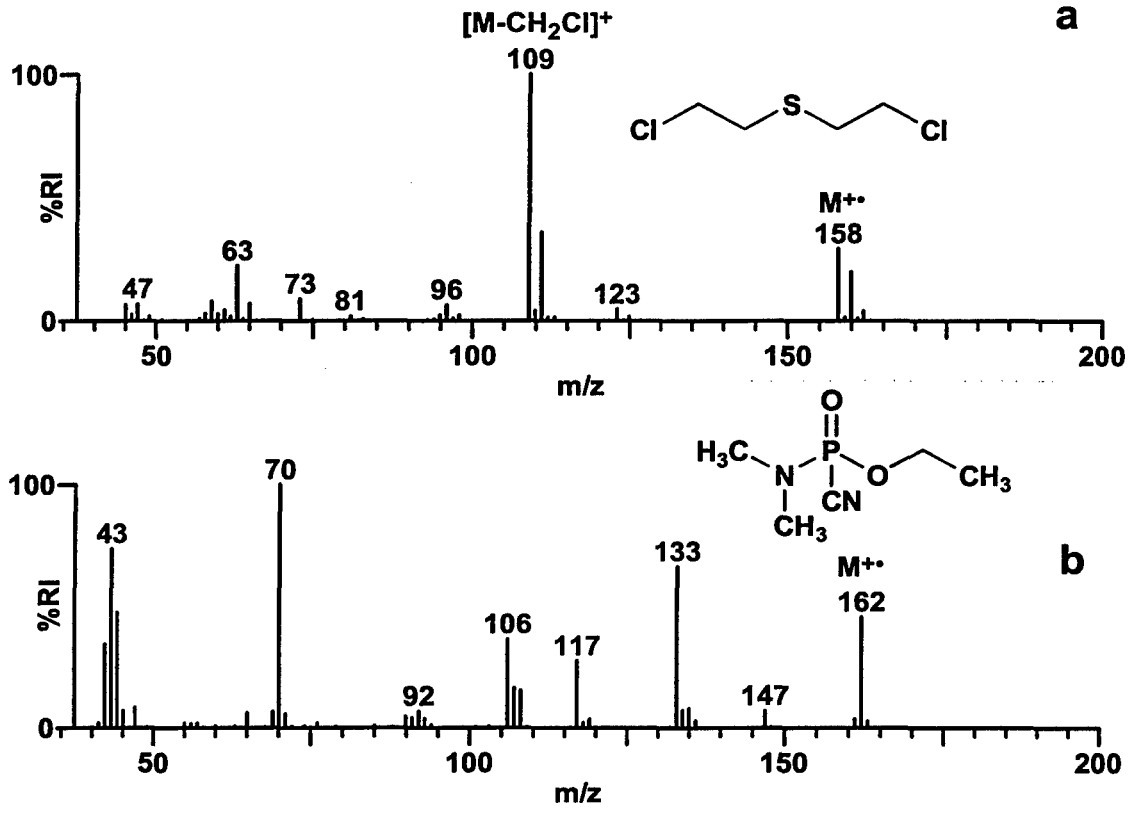

Figure 10. *EI-MS data acquired for a) H and b) GA during GC-MS analysis.*

GC-MS analysis of soil sample extracts

Two different contaminated soil samples were provided as unknowns for GC-MS analysis. Each of the soils and its corresponding control were extracted with dichloromethane using the method described in the Experimental. Sample extracts (1 μL) were analysed by GC-MS and the acquired mass spectra were interpreted and compared to library spectra contained in the EI database supplied with the GC-MS instrument. Figure 11 illustrates the GC-MS chromatograms obtained for the dichloromethane extracts of the soil samples spiked with GF and HQ, a munitions grade mustard sample. The GF spiked soil samples contained only GF and a small amount of phthalate. The HQ sample was more complex, containing H, sesquimustard (Q) and a number of minor related compounds. GC retention time and acquired EI-MS data for GF and H were similar to those acquired during test mixture analysis, while the EI data acquired for Q (and other compounds) was similar to library mass spectra contained in the EI database supplied with the GC-MS instrument. Figure 12 illustrates a typical library match between acquired and library data for Q.

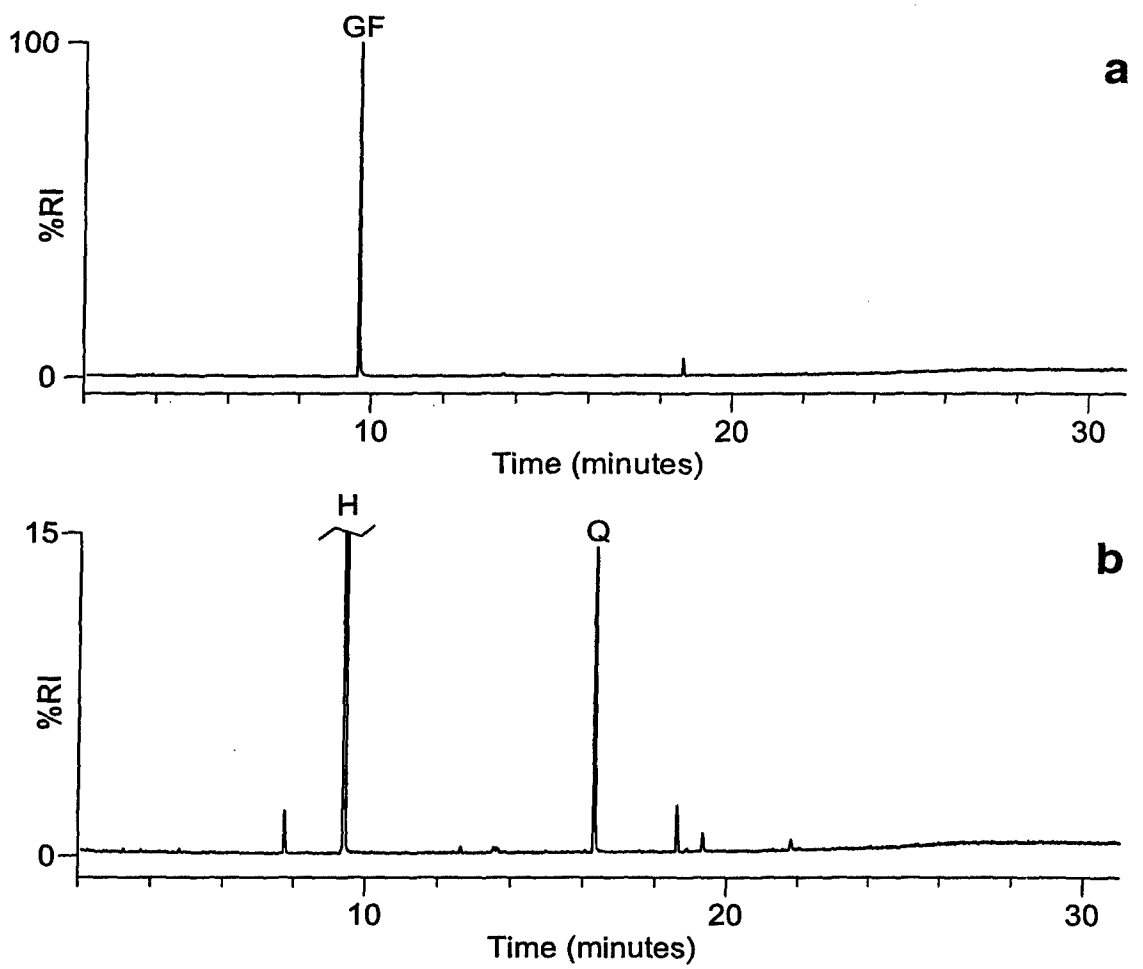

Figure 11. *GC-MS total-ion-current chromatograms obtained for the dichloromethane extracts of the soil samples spiked at the 50 µg/g level with a) GF and b) HQ.*

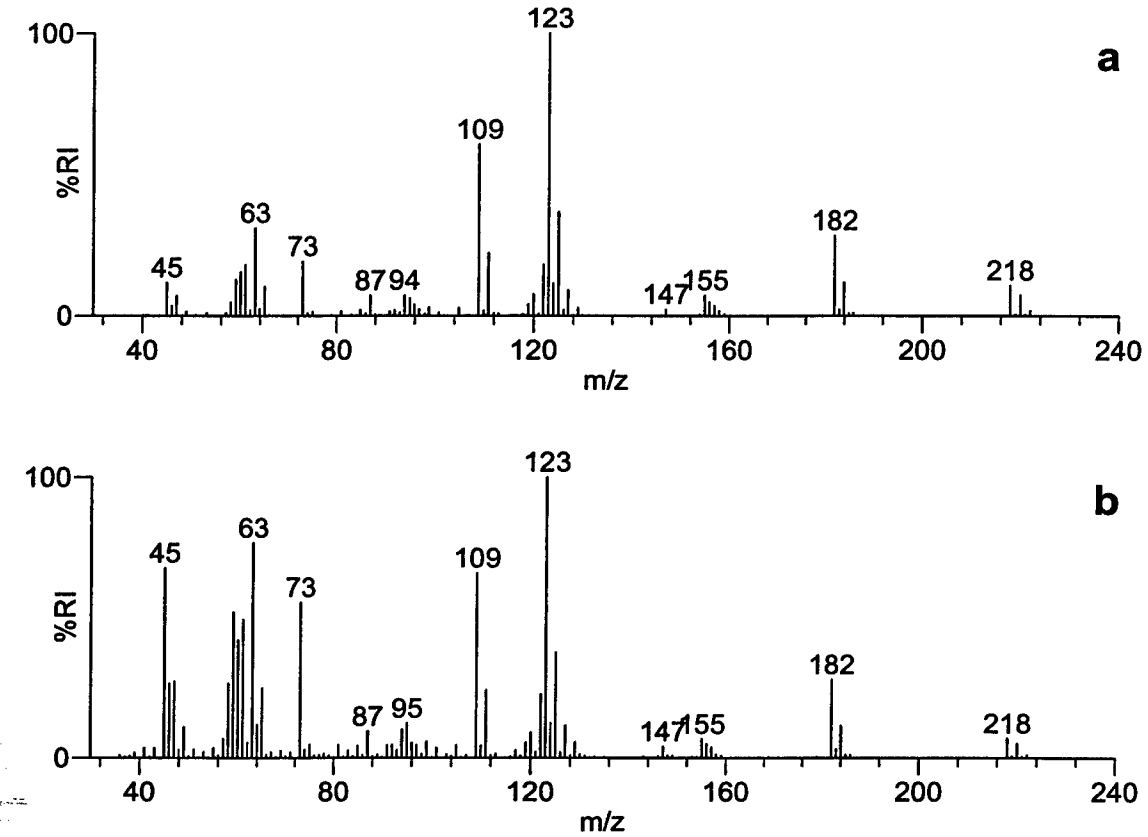

Figure 12. *a) EI-MS data acquired for Q during analysis of soil sample extract. b) EI-MS data contained in the EI database supplied with the GC-MS instrument.*

Chemical warfare agent detection devices

The Canadian Forces (CF) have six in-service devices for the detection of chemical warfare chemical warfare agents. These devices are based on a number of different chemical principles ranging from chemical solubility to ion-mobility spectrometry and detect the presence of a range of nerve, blister, blood and choking chemical warfare agents.

3-Way Paper is a dye-impregnated paper that can detect the presence of nerve and blister agents in their liquid state. Dye-solubility is the principle behind 3-Way Paper and a positive response to agent would be indicated by the appearance of a spot with the corresponding dye color. G-type nerve agents are visualized by a yellow dye, V-type nerve agents by a green dye and blister agents by a red dye. Available in booklet form with a legend on the cover for colour comparison, 3-Way Paper is produced and marketed by Anachemia Canada Inc. It's ease of use and low cost make the 3-Way Paper an economical tool in the detection of chemical warfare agents.

The Nerve Agent Vapour Detector (NAVD) is a small clear plastic ticket with two paper sections, one impregnated with acetylcholinesterase and the other impregnated with a

colorless dye that reacts with active acetylcholinesterase to form a blue complex. Once the paper sections are wetted and exposed to the suspect atmosphere, they are pressed together. In the absence of nerve agent, a blue-coloured spot develops but if nerve agent is present, the enzyme is inhibited and no blue spot appears. The NAVD is highly selective but it is limited to nerve agent detection and does not indicate which nerve agent is present. It's 5.5 x 2.5 x 0.2 cm size makes it the smallest detection device in-service with the CF. It is manufactured and distributed by Anachemia Canada Inc.

The M256A1 kit was developed in an attempt to combine the detection of many chemical warfare agents in a single device. This kit, produced by Anachemia Canada Inc., contains a hard-plastic carrying case, 12 sampler-detectors and detailed instruction cards that are attached to the M256A1 case. Each sampler-detector incorporates an enzyme impregnated paper spot for nerve agent detection, as described for the NAVD; a test spot, and accompanying heater assembly, for blister agents such as mustard (H) and phosgene oxime (CX); a tablet to identify the presence of Lewisite (L); and finally a test spot for blood agents such as cyanogen chloride (CK) and hydrogen cyanide (AC). Small chemical-filled ampoules are broken to allow chemical combinations to flow through plastic channels and wet the appropriate test spots prior to a 10 minute vapour exposure. Instructions for use are also provided on each sampler-detector's protective foil wrap. This detector system is small, inexpensive and allows users to determine whether their immediate environment is safe enough to remove their protective gear.

The Chemical Agent Monitor (CAM) has been an integral part of the Canadian Forces chemical warfare agent detection equipment since 1986. The CAM was initially developed and produced by Graseby Dynamics Ltd. in the UK. Graseby Dynamics Ltd. has since been incorporated into Smiths Detection who now holds the rights to produce and market the complete line of CAM products. The CAM uses ion-mobility spectrometry (IMS) to detect nerve and blister agent vapours. Air samples are drawn into the nozzle (i.e. probe) and pass through a silicon membrane before coming into contact with acetone vapour, provided by the sieve breather assembly, circulating in the CAM. The green arrows in Figure 13 illustrate the internal airflow pattern. The acetone and agent molecules are ionized by the Ni^{63} radioactive source to form low-mobility ion clusters. The gating grid is opened to allow the ion clusters to travel towards the ion collector plate, which maintains positive or negative polarity depending on the operator's choice of G or H mode. This process is repeated many times per second and the time it takes for the clusters to reach the collector plate, referenced to an internal reactant ion peak drift, is compared to known times for agent ion clusters. If the measured ion-mobilities correspond to known agent ion mobilities, a bar graph response is produced on the LCD graphical display. The amount of ions measured is relative to the concentration of agent in the sampled vapour and the number of bars visualized, from one to eight, reflects the estimated concentration. This method of detection provides some selectivity for chemical warfare agents by monitoring only those times in the IMS spectrum where nerve or blister agent ion clusters appear. This programmed selectivity can prevent the user from observing high concentrations of toxic chemicals not included in the manufacturer's software. Newer versions of the CAM, which include the CAM2Plus and the ECAM, still detect the classical chemical warfare agents (H series, G series and VX) but are also programmed to include the blood and choking agents AC, phosgene (CG) and chlorine (Cl_2). While in operation, the CAM samples continuously and responds to low levels of agent in one to five seconds. The size (1.9 kg) and cost of a CAM is significantly larger than the three other personal chemical warfare agent detectors used by the CF but none of these provide such rapid detection as the CAM.

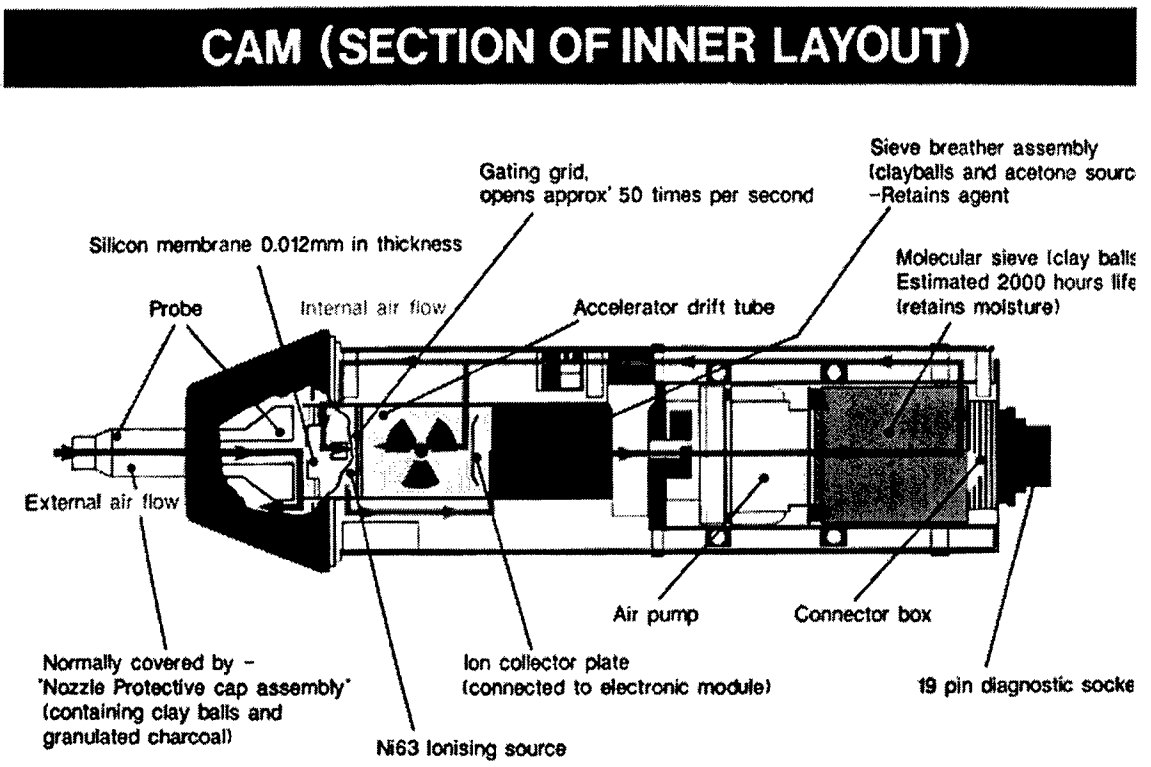

Figure 13. *General schematic for the Chemical Agent Monitor (CAM).*

A system for simultaneous remote monitoring of G and H agents was required by the CF during the Iraq War in 1990-91 and DRDC Suffield developed the CADS II to fulfill that requirement. This networked system uses two CAMs per sensor station, one operating in G mode with the other in H mode. A solar cell with back-up battery provides power at each station and a central control unit controls the network of stations, each of which may be deployed up to 3000 meters from the central control unit.

The only detection system currently in-service with the CF that is specifically engineered for simultaneous G and H monitoring, is the GID-3, an IMS-based detector now being manufactured by Smiths Detection. The unique feature of the GID-3 is the dual ion drift tubes and collector plates that allow for both positive and negative ions to be measured simultaneously. This system is fitted with a much larger battery than the CAM and is well suited for unmanned operation or vehicle mounting. The GID-3 has a bar graph for visual alert, audible alarms and can be remotely monitored through a networked warning system.

The CF is currently evaluating commercially available chemical warfare agent detection equipment to increase their capability in this area. Some of the equipment of interest include the AP2C (and TIMs) from Proengin in France, the HAPSITE manufactured by Inficon in the US and the M-90 manufactured by Environics Oy in Finland. The AP2C and TIMs detectors are based on flame photometry, the HAPSITE is a man-portable gas chromatograph with a mass selective detector (GC-MS) and the M-90 is a modified IMS system. DRDC Suffield also has research underway to examine and extend the use of solid-phase micro-extraction (SPME) techniques in conjunction with field-portable GC-MS for the rapid detection and identification of chemical warfare agents and toxic industrial chemicals (TICs) that may be encountered by the CF and civilian first-responders.

Conclusions

Each of the analytical exercise participants conducts sample handling and analysis for a variety of target compounds for their government departments (Health Canada, Canadian Food Inspection Agency, Royal Canadian Mounted Police and Environment Canada). If their sample handling methods co-extracted chemical warfare agents the analysts would be able to identify the common chemical warfare agents, provided GC-MS analyses were conducted under full scanning EI-MS conditions.

The analytical exercise participants successfully analysed a chemical warfare agent test mixture by GC-MS, interpreted the acquired mass spectra and correctly identified the chemical warfare agents spiked into two unknown soil samples by GC-MS. Chemical warfare agents were identified in the soil sample extracts on the basis of both a GC retention time and EI mass spectrometric match with authentic reference standards (or library data).

The analytical participants were briefed on both safety considerations and chemical warfare agent detection devices. Detection devices, including the Chemical Agent Monitor, were demonstrated and handled by the participants and several sampling kits were opened up for examination.

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(U) The Chemical Cluster, one of three clusters created by the Chemical, biological, radiological and nuclear Research and Technology Initiative (CRTI), was established to help Canada prepare for possible terrorist events. This working group, made up of representatives from Canadian government departments, has identified a number of chemicals of concern and assigned laboratories with appropriate expertise to provide the analytical support necessary to confirm these compounds in suspect samples. The Royal Canadian Mounted Police (RCMP), in its lead forensics role, will attempt to tentatively identify the chemical(s) of concern and pass on the samples to the responsible laboratory within the Chemical Cluster. Samples containing large amounts of relatively pure chemical warfare agents should trigger a response with one the chemical monitoring devices (e.g., Chemical Agent Monitor) used by the RCMP to triage samples. Defence R&D Canada – Suffield (DRDC Suffield) has been tasked to analyse samples suspected to contain chemical warfare agents for the Chemical Cluster and would receive this type of suspect sample. There remains a possibility that samples with a lower level of chemical warfare agent contamination might inadvertently find their way into a laboratory tasked with another type of analysis. To manage this possibility, the laboratories receiving these types of samples should have an analytical screening capability to allow for the tentative identification of chemical warfare agents in samples and sample extracts. This report summarizes the chemical warfare agent training course in sample preparation and analysis by gas chromatography-mass spectrometry (GC-MS) given by DRDC Suffield to other Chemical Cluster laboratories.

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